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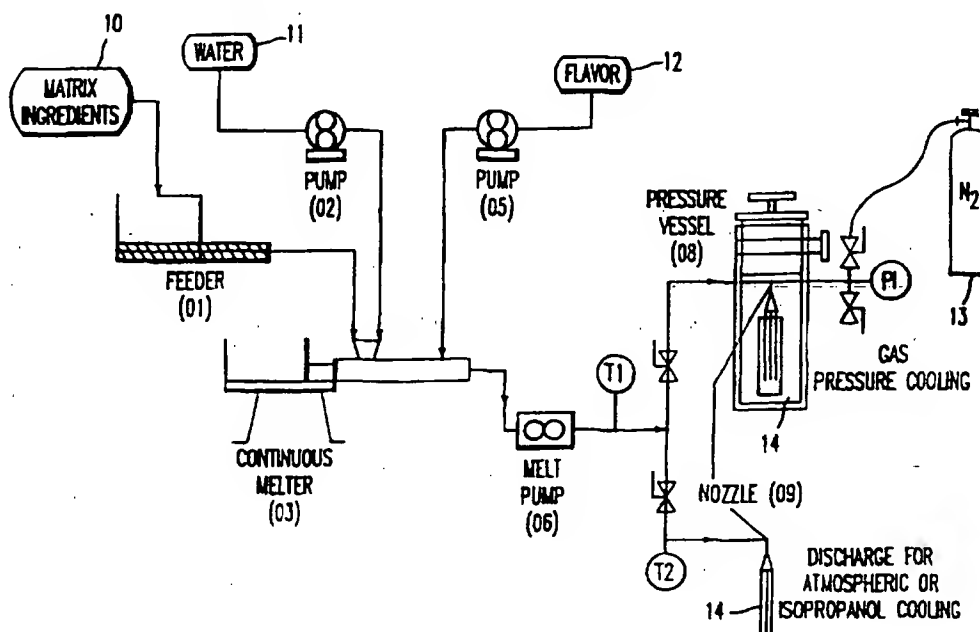
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## (54) Title: FLAVOR ENCAPSULATION



## (57) Abstract

A process for incorporating a volatile component into a matrix comprising: (a) forming a melt comprising said volatile component (12) and said matrix (10), (b) solidifying said melt under a pressure sufficient to prevent substantial volatilization of said volatile component (8). In a preferred embodiment, the volatile component is a volatile aroma compound such as those contained in coffee grinder gas or emitted when baking dough or roasting a food.

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TITLE OF THE INVENTION

## FLAVOR ENCAPSULATION

This is a continuation-in-part of U.S. Patent Application  
5 Serial No. 07/948,437, filed September 22, 1992, which is  
incorporated herein in its entirety by reference.

BACKGROUND OF THE INVENTIONField of the Invention:

The present invention relates to techniques to encapsulate  
10 materials which can undergo compositional changes in process  
and/or storage. Such encapsulation improves material shelf-life  
and usefulness in the preparation of products such as foods. The  
present invention also relates to a method for encapsulating  
coffee aroma compounds, such as those contained in natural or  
15 synthetic coffee grinder gas, in a glassy matrix of a food  
polymer as well as compositions in which such coffee aroma  
compounds are so encapsulated.

Discussion of the Background:

It has long been recognized that it is desirable to  
20 encapsulate materials so as to protect them from volatilization,  
the degradation effects of oxygen and heat, moisture, internal

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and external molecular interactions and the like. Flavors are complex substances made up of multiple chemical components, some comparatively stable, some extremely volatile, others unstable to oxidation and reactive interactions and the like. Many  
5 flavorants contain top notes (i.e., dimethyl sulfide, acetaldehyde), which are quite volatile, vaporizing at or below room temperature. These top notes are often what give foods their fresh flavors.

Numerous techniques have been suggested and many  
10 commercialized for the encapsulation of flavors. However, all of these techniques suffer from one or more deficiencies. One of the most common techniques for encapsulating flavorants is spray drying. While this process directly produces a finely divided product which can be readily handled and used in the preparation  
15 of finished foods, spray drying suffers from several serious deficiencies. First, it is difficult to incorporate top notes into spray dried flavorants in an efficient manner. Inherent in spray drying is the loss of volatile materials. Furthermore, materials which are heat and/or oxygen sensitive are adversely  
20 affected by spray drying. The effect of heat, oxygen and volatilization can make a substantial change in the materials' composition, which in turn results in undesirable changes in flavor characteristics.

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Freeze-drying solutions of matrix materials containing either dissolved or dispersed flavors has also been used to produce encapsulated flavors. These methods generally result in losses of highly volatile components, and products having a foamy, porous structure.

Yet another technique which has been employed is that of melt encapsulation of materials in carbohydrate matrices. In this application a carbohydrate melt is prepared and the encapsulate is added. The resulting solution is introduced into a quenching medium to produce a solid carbohydrate product containing the flavor. This technique while successful, is again, limited to comparatively high boiling point flavors because the carbohydrate solution is produced and delivered to the quenching medium at elevated temperatures. This technique inherently can result in the loss of some of the low boiling point constituents in the flavor. Because of such losses, it is common to enhance the flavorant by adding extra low-boiling components. The conventional quenching agent which is commercially employed is isopropyl alcohol. The traces of the isopropyl alcohol remaining in the product after quenching can be detrimental. This technique limits the materials which can be encapsulated to those which are immiscible in the matrix. An additional disadvantage of the product resulting from this process is that although reasonably dense, the product may

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contain microporosity when low boiling point components are present in the flavor. The microporosity increases the surface area, and thus, may increase the evaporation of volatiles and the potential for degradation of the product by interaction with atmospheric oxygen. Furthermore, the effect of the microporosity is enhanced as the product is sold in a finely divided state, which increases the surface area of the particles and thus the possibility that degradation of the flavor will occur if the product is stored over a period of time.

10       The above encapsulation technology was first developed using batch type melting and mixing equipment. These techniques have been improved as described in U.S. Patents 4,610,890 ('890) and 4,707,367 ('367). In these patents, a process is described for preparing a solid, essential-oil containing composition. This composition is prepared by forming an aqueous, high-solids solution containing a sugar, a starch hydrolysate and an emulsifier. The essential oil is blended with this aqueous solution in a closed vessel under controlled pressure conditions to form a homogenous melt which is then extruded into a relatively cold solvent, normally isopropanol, dried and combined with an anti-caking agent after grinding. A discussion of these and other prior art techniques for encapsulating materials can be found in U.S. Patent 5,009,900. The patents '890 and '367 suffer from the same deficiencies noted in prior art techniques, i.e.,

15  
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loss of volatile compounds and limitations to immiscible flavor encapsulates.

While the above described solidified melt encapsulation technology was first developed using batch type equipment, more recently similar continuous processes have used extruders to produce encapsulated products. One problem encountered in extrusion is the difficulty in obtaining an encapsulant which will melt under reasonable extrusion temperatures. An additional problem with extruded products under typical melting temperatures is that the product will expand and foam upon exit from the extruder head due to expansion of contained volatiles. The objective in encapsulation is to form a hard, dense, glassy type encapsulant. One approach is that described in U.S. Patent 4,820,534 ('534). This patent suggests utilizing as the encapsulant a mixture of two materials, one having a high molecular weight and the other having a low molecular weight; as a result, the mixture may be successfully extruded. During extrusion, according to '534, the minor component melts and the major component dissolves into the minor component. The volatile flavorant becomes dispersed or solubilized within the molten mass which upon cooling produces a single phase matrix. In order for volatile components to be retained, and expansion of the matrix prevented, it is necessary in the process of '534 to minimize the temperature at the extruder head. If the material exits the



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extruder at a higher temperature, volatiles will be lost from the mixture. The '534 technique needs to utilize as the encapsulant a mixture of materials, one having a melting point sufficiently low such that the remainder will melt into it thereby becoming extrudable under reasonable process conditions.

U.S. Patent 5,009,900 ('900) is directed to a procedure very similar to that of '534 only using a more complex mixture of materials to form the encapsulant material. The '900 patent requires a water-soluble, chemically-modified starch, maltodextrin, corn syrup solids and mono- or disaccharides. The flavorant is mixed into the mixture and the result is extruded.

It would not be possible with either of the techniques of '534 or '900 to encapsulate pure low boiling point materials such as acetaldehyde in a dense matrix at commercially significant loads since the resulting product would foam due to the vaporization of acetaldehyde as it exits the extruder. Furthermore, in both techniques one is restrained by processing considerations in the selection of encapsulate material. Similar techniques are taught in U.S. Patent 4,232,047 ('047). The process of '047 proposes to encapsulate a seasoning or flavoring such as oleoresin, essential oils and the like in a matrix of starch, protein, flour and the like. This technique involves the use of extrusion under high pressure. However, like the other techniques, it is limited in the materials which can be used as

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the encapsulating agent and the materials to be encapsulated therein. The temperatures involved could cause the loss of volatile top notes.

Another example of the technology which is available is U.S. Patent 4,689,235 ('235) which like '900 and '534 is directed to specific matrix materials for use in encapsulation. This patent relies upon the use of an emulsifier to achieve success.

As evidenced by the foregoing patents, significant effort has been expended in attempting to develop a successful method for encapsulating volatile and/or unstable flavors using solidified melts. These techniques would have the advantage over spray drying in that the product, if a dense matrix can be formed, would not be porous like the spray dried product, thus the flavor encapsulate would be more stable. It would be anticipated that such products would have a long shelf life. However, these technologies do not assure a non-porous product when the pressurized melt exits to ambient pressure and temperature.

In addition to the foregoing deficiencies which have been noted in the prior art techniques, still other deficiencies are that each of these processes is very specific to the encapsulating composition. That is, they significantly restrict the compositions which can be used as encapsulants to a very narrow range.

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In producing encapsulated products, it is desirable that the encapsulant have a softening temperature significantly above room temperature. If the softening temperature is low, the material will become tacky, forming lumps which are difficult to handle and process. Patents '534 and '900 suggest utilizing complex mixtures of materials as the encapsulant, such that the resultant matrix is in the glassy state with softening temperatures greater than 40°C.

While solidified melt techniques have, to greater or lesser extent, been utilized commercially to encapsulate some flavorants in dense amorphous matrices, there are many flavorants which simply cannot be encapsulated by existing technology. For example, flavorants which are normally commercially produced in the form of a solution simply cannot be encapsulated at useful levels using existing techniques if the solvent plasticizes the matrix materials. With flavorants such as vanilla extract, it is impossible to remove the water/alcohol solvent without adversely affecting the properties of the vanilla. Even in concentrated form, there still would be appreciable solvent present. Accordingly, vanilla extract has not been successfully encapsulated at commercially useful levels using the above techniques. Therefore, a need exists for a new process to produce dense, non-porous matrices to encapsulate materials that exist in high concentrations of solvents.

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A long-standing problem in the instant coffee art is the preparation of instant coffees having the same aroma as freshly roasted and ground coffee itself. A similar problem of aroma dissipation also exists in holding ground roast coffee for a time before consumption. Many methods have been utilized in attempting to approach this desired end result. For example, aroma materials have been stripped from roast and ground coffee and added back to instant coffee after complete processing; and coffee oil collected from extracted roast/ground coffee beans has been used to absorb the aroma volatiles and added back to final instant coffee product as an aroma-enhancing material. Other techniques which have commonly been employed include dry distillation of coffee beans to obtain aroma materials, utilization of complicated extraction processes in order to obtain coffee aroma materials, and the like. One method disclosed in the prior art is to obtain an aroma frost by low temperature condensation methods. Aroma frost is a low temperature condensate of volatiles which escape during coffee processing, for example, during roasting, grinding, steam distillation, extracting where it is employed, and dry distillation and the like. These volatile constituents are often obtained by low temperature condensation of escaping gaseous material, for example at liquid nitrogen temperatures, by passing the escaping gas through a liquid nitrogen trap to yield a

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condensate which is referred to herein as an aroma frost. The aroma frost may be grinder gas frost, roaster gas frost, a dry distillation frost, a steam distillation frost, and the like.

However, once obtained, to be of value aroma frost must then be incorporated in a relatively stable manner into the coffee product whose roast and ground coffee aroma is desired to be increased. Typically, this coffee product is an instant coffee because instant coffees are notably deficient in characteristic roast and ground coffee aroma. Additionally, the aroma frost itself must be incorporated into a stable carrier in order to have any significant amount of retained aroma value. The most common carrier is coffee oil.

Grinder gas, that is the gas which is released from roasted whole coffee beans when their internal cell structure is disrupted, such as during grinding of the beans and which also continues to be evolved from the disrupted and/or fractured beans for a short period thereafter, has long been recognized in the art as a highly desirable natural coffee aroma. A great deal of effort has been directed toward the development of a method for recovering and fixing a high percentage of grinder gas aromas on a substrate for subsequent addition to coffee products, particularly soluble coffee powder.

U.S. Patent no. 3,021,218 discloses a method whereby a coffee aroma condensate is vaporized to admit the more volatile

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coffee aromas into void spaces of a container filled with soluble coffee. U.S. Patent no. 2,306,061 describes the addition of condensed grinder gas to chilled soluble coffee powder. These two methods are similar in that grinder gas aromas are contacted  
5 directly with coffee particles in order to improve the aromatics of the coffee product. However, both methods fail to provide the convenience, stability, and high level of grinder gas fixation, desired in the coffee field.

Several methods for fixing grinder gas aromatics in a  
10 glyceride substrate are known in the art, such glycerides as coffee oil, bland-tasting vegetable oils, and triacetin being especially useful for this purpose. U.S. Patent no. 3,939,291 teaches contacting condensed grinder gas and a glyceride in a pressure vessel at a temperature above the critical temperature  
15 of liquid carbon dioxide, then slowly venting the vessel while maintaining the temperature above carbon dioxide's critical temperature. This method effectively prevents the formation of liquid carbon dioxide at any point during the glyceride aromatization procedure.

20 U.S. Patent no. 3,979,528 describes contacting a glyceride and condensed grinder gas in a pressure vessel at a temperature of 70° to 75°F, and a pressure above 100 psig to enable aromatic transfer from the liquid carbon dioxide phase to the liquid glyceride phase, and then venting the vessel. This procedure

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involves repeated venting of said pressure vessel from a high pressure ranging from 75 to 120 psig, to a low pressure of 0 psig.

U.S. Patent no. 4,007,291 discloses contacting a glyceride with condensed grinder gas in a pressure vessel at a temperature of 70° to 85°F, and a pressure in excess of 700 psig, then slowly venting the pressure vessel, preferably isothermally. U.S. Patent no. 4,119,736 discloses the removal of a water phase from a pressure vessel containing condensed grinder gas at a high pressure and temperature, contacting the demoinsturized grinder gas with a glyceride, and slowly venting the pressure vessel. The separation of aromas from the removed water phase, such as by vacuum distillation, and to reclaim or recycle vented aromatics, is also described.

European patent application 205,204 describes a complex process for the preparation of a liquid coffee aroma, which comprises first condensing the coffee grinder gas, then ~~subjecting it to a high pressure, and finally, through a number~~ of further operations, converting it into a liquid coffee aroma substantially free from CO<sub>2</sub>. This coffee aroma can be used for aromatizing a substrate, such as ground coffee or a coffee powder.

A similar process is described in European patent application 213,247 which additionally discloses the addition of

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coffee oil during one of the processing stages, so that ultimately an aromatized coffee oil is obtained, which can be combined with soluble coffee powder.

European patent application 201,968 describes the treatment  
5 of ground coffee with a carrier gas to remove aroma components from ground coffee. The aroma components can then be removed from the carrier gas by condensation. European patent application 144,758 discloses a process in which ground coffee is heated to a temperature between 30° and 95°C, and the released  
10 aroma components are contacted with instant coffee.

European patent application 28,043 describes aromatizing coffee, by cooling coffee grinder gases with dry ice, optionally together with the coffee. European patent application 41,370 also describes the condensation of coffee aroma components on  
15 coffee at cryogenic temperatures. British patent publication 2,063,640 also teaches the condensation of aroma components on ground coffee.

U.S. Patent no. 3,823,241 describes a method wherein an absorbent carrier is cooled to at least - 40°F. (preferably -  
20 150°F.) and then placed in communication with roast and ground coffee under pressure conditions which transfer the coffee aroma to the absorbent carrier. Another example is aroma frost equilibration with an aroma substrate which is typically a liquid glyceride such as coffee oil. U.S. Patent no. 3,783,163



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discloses a method for aromatizing edible oils by adding the oil to a cryogenic fluid to form a slurry, adding an aroma frost to the slurry, preferably with mixing, and then allowing the mixture to equilibrate to evaporate the cryogenic fluid, leaving behind a residue of aroma-enhanced oil.

U.S. Patent no. 4,335,149 and European patent application 10,804 disclose methods of condensing grinder gas on a food substrate such as ground coffee at cryogenic temperatures.

U.S. Patent no. 4,520,033 describes a method for preparing capsules which contain a foamed core containing an aqueous essence such as coffee distillates. The core may be foamed with coffee grinder gas.

U.S. Patent no. 3,821,447 discloses a method of stabilizing coffee aromas by condensing grinder gas with an edible glyceride such as an oil and then removing excess water. The resulting blend may then be mixed with an aqueous coffee extract or dry soluble coffee.

U.S. Patent no. 3,991,223 teaches a method for treating steam coffee aromas by means of liquid glyceride extraction to yield glyceride and aqueous phases with smooth, buttery flavors.

U.S. Patent no. 4,044,167 teaches a method of aromatizing soluble coffee products in which grinder gas is added to a liquid glyceride which is then frozen and then blended with the soluble coffee.

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U.S. Patent no. 4,556,575 discloses a method for aromatizing soluble coffee by contacting the water phase, normally drained from equilibrated grinder gas and discarded, with a glyceride to recover coffee aromatics in the glyceride which may then be  
5 refluxed with liquid carbon dioxide before combining with a soluble coffee powder.

U.S. Patent no. 5,079,026 describes a process for preparing an expanded gasified coffee glass in which a melt of coffee solids and coffee oil is injected with a gas such as nitrogen or  
10 carbon dioxide, extruded through an orifice, allowed to expand, and then quickly cooled.

U.S. Patent no. 5,035,908 discloses a method for preparing a coffee glass by evaporating a coffee extract to form a viscoelastic fluid and rapidly cooling the fluid. The hot  
15 viscoelastic fluid may be gasified to lower the density of the glass, in which case the gasified fluid is allowed to expand prior to cooling.

European patent application 353,806 teaches a method in which the content of the aroma components in coffee grinder gas  
20 is increased, such as by increasing the pressure or removing a portion of the CO<sub>2</sub>, before contacting with coffee.

Synthetic coffee aroma, such as those described in U.S. Patent nos. 1,696,419 3,852,481, 3,873,746, 3,655,397, and 4,378,380 have also been employed to enhance overall coffee

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flavor and aroma by mixing together compounds known to exist in coffee.

However, none of these techniques is completely satisfactory with regard to providing a composition which exhibits a prolonged retention of the highly volatile aroma associated with freshly  
5 ground coffee. Thus, there remains a need for a method of adding coffee aroma compounds, such as those contained in coffee grinder gas, to a coffee composition which results in the stable retention of the coffee aroma compounds.

#### DESCRIPTION OF THE INVENTION

Accordingly, it is an object of the present invention to provide a process to encapsulate a wide range of materials, including flavorants, fragrances, colors, pharmaceuticals and the like, without the loss of volatile materials, especially gases,  
15 oxidative degradation, molecular reactions and other adverse interactions with the environment.

Further, it is another object of the present invention to provide a process for encapsulating both miscible and immiscible materials.

20 It is yet another object of the present invention to provide an encapsulating system for flavorants which are normally dissolved in water, alcohol or other volatile solvent systems.

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It is still a further object of this invention to provide a technique for encapsulating flavor components which have low boiling points in a dense non-porous encapsulant.

5 It is still another object of the present invention to provide a process which allows the use of encapsulating materials which would normally puff or foam when the melt is released from pressure.

10 It is also an object of the invention to prevent molecular migration by the formation of the dense amorphous solid, thus reducing molecular interactions and changes.

It is another object of the present invention to provide a method for preparing compositions which contain coffee aroma compounds, such as those contained in coffee grinder gas.

15 It is another object of the present invention to provide a method for preparing compositions which contain coffee aroma compounds, such as those contained in coffee grinder gas, and retain these compounds for prolonged periods of time.

20 It is another object of the present invention to provide a method for preparing compositions in which coffee aroma compounds, such as those contained in coffee grinder gas, are stably encapsulated.

It is another object of the present invention to provide novel compositions which contain coffee aroma compounds, such as those contained in coffee grinder gas.

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It is another object of the present invention to provide compositions which contain coffee aroma compounds, such as those contained in coffee grinder gas, and retain these compounds for prolonged periods of time.

5 It is another object of the present invention to provide compositions in which coffee aroma compounds, such as those contained in coffee grinder gas, are stably encapsulated.

10 It is another object of the present invention to provide novel compositions which contain other volatile aroma gases, such as those emitted when baking dough products, like breads, or roasting foods.

15 It is another object of the present invention to provide compositions which contain volatile aroma gases such as those emitted when baking dough products, like breads, or roasting foods and retain these volatile aroma gases for prolonged periods of time.

20 It is another object of the present invention to provide compositions in which volatile aroma gases such as those emitted when baking dough products, like breads, or roasting foods, are stably encapsulated.

It is another object of the present invention to provide a method for preparing compositions which contain volatile aroma gases such as those emitted when baking dough products, like bread, or roasting foods.

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These and other objects of the invention which will become apparent from the description hereafter, have been achieved by a process wherein a melt is made of the encapsulant and encapsulate; and the molten matrix containing the encapsulate is  
5 cooled by overriding solid, liquid, or gaseous pressure into a dense amorphous matrix.

A second embodiment involves forming a melt containing an encapsulate dissolved in a solvent and an encapsulating matrix which is optionally subjected to an elevated pressure, followed  
10 by venting to remove at least some of the solvent while largely retaining the encapsulate in the product.

In this invention, the dense amorphous, essentially non-crystalline solid encapsulant may be described in many cases but not exclusively by those knowledgeable in the art as a 'glass' as  
15 characterized by a glass transition temperature.

In a preferred embodiment, the present invention provides a process in which the encapsulate contains a volatile coffee aroma compound, such as one or more components of natural or synthetic coffee grinder gas. In another preferred embodiment the  
20 encapsulate is a volatile gas such as those emitted when baking dough products, like bread, or roasting foods, like nuts, or peanuts, or meats.

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BRIEF DESCRIPTION OF THE DRAWINGS

A more complete appreciation of the invention and many of the attendant advantages thereof will be readily obtained as the same become better understood by reference to the following  
5 detailed description when considered in connection with the accompanying drawings, wherein:

Figure 1 is an illustration of the present process where the flavor component to be encapsulated is introduced into the extruder where a matrix material has been melted. The drawing  
10 shows both atmospheric and pressurized discharge points. These were used in examples to produce comparative samples;

Figure 2 is an illustration of another embodiment wherein the matrix is first melted in an extruder and the flavor and melted matrix material are mixed in a static mixture and then  
15 recovered. The drawing shows both atmospheric and pressurized discharge points. These were used in examples to produce comparative samples;

Figure 3 is an illustration of the present process where the flavor component is diluted in a volatile solvent and said  
20 solvent is removed via venting; and

Figure 4 is a generalized overview of process sequence steps which can be utilized in the present process.

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DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the present process, melting equipment (herein referred to as "melter") is utilized to convert the matrix from solid to liquid form. The components of the matrix are introduced into a melter where they are liquefied. The melting may be accomplished in a batch containment. The melter also can be simply a device transporting the matrix through a heating zone wherein sufficient heat is introduced to convert the matrix to liquid form, i.e., melted. The process can utilize a conventional single or twin screw extruder having mixing zones, homogenizing zones, melting zones, venting zones and the like as is conventionally known in the art. The matrix materials may be composed of a variety of melting compositions so that the resulting dense matrix will not become sticky and agglomerate at lower temperatures yet will melt/dissolve at under normal application conditions and temperatures as described in the prior art. Any meltable matrix ingredient can be utilized.

When utilizing materials having a low melting temperature, it is often possible to directly melt the material in a suitable processor. As described in the art, it may be necessary with high melting temperature materials to utilize a solvent with the purpose of generating enough "plasticity" to the matrix materials so they can be processed successfully. The amount of solvent added generally is insufficient to dissolve all of the matrix



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materials, but is sufficient only to increase plasticity. The minimum amount of solvent is utilized which provides enough plasticity to the matrix ingredients such that they can be successfully processed. The optimum amount of solvent for use  
5 varies from matrix to matrix.

The solvents which can function as the plasticizer include any liquid material in which the matrix is soluble. Typical solvents include water, water-ethanol, glycerin, propylene glycol and the like. An optional process step, venting, can be added  
10 where some or all of the solvent can be removed. Following, the encapsulate is then mixed into the matrix. Essentially any encapsulate, insoluble, slightly soluble or miscible in the matrix may be employed in this particular embodiment. In cases where the encapsulate exists as a solution in a volatile solvent  
15 (e.g. water, alcohol), the melt may be vented to substantially eliminate the encapsulate solvent.

Cooling of the melt can be accomplished at ambient conditions, with cooled gas, by direct contact with metal belts or rolls, or by quenching in a suitable solvent, as in the prior  
20 art, or most preferably as introduced by the invention, under pressure so as to prevent "puffing" or expansion of the matrix material into a non-dense, porous form.

When one is concerned with either reducing the microporosity of the matrix or with encapsulating volatile components, this

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embodiment can be performed using a wide variety of apparatus to form the melt and to extrude it through a die into the pressurized zone. The simplest technique is to form a melt using the procedures described in U.S. Patents 4,610,890 and 4,707,367.

5 These techniques utilize a batch reactor to form the melt. In this technique, the matrix material with suitable solvent is introduced into the tank and melted. Once the melt has been established, then the material to be encapsulated is added. It is possible to vary this procedure where the material to be  
10 encapsulated also functions as a solvent for the solid matrix material. In this instance, the encapsulate and solid matrix are added together without the use of any separate solvent and the melt established. The tank or vessel in which this is accomplished, can either be opened to the atmosphere or closed.

15 It is particularly preferred that the vessel be a pressure vessel and closed during the process so as to reduce the losses of any volatile components in the material to be encapsulated. If the volatile components comprise a significant portion of the encapsulant, then pressure should be established in the vessel so  
20 as to reduce the vaporization of the low boiling components in the vessel and thereby increase their yield. Once the melt has been established, the vessel can then be pressurized further, if necessary, and the pressure in the vessel used to force the melt through the die into a solidification zone. Prior art as

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described above used an ambient pressure solidification step. The present invention introduces the use of a pressurized solidification zone having a pressure sufficient to preclude the vaporization of the significant portion of the volatile components in the melt during solidification. The pressure in the solidification zone is chosen to be sufficient so as to prevent puffing or microporosity. The melt can be delivered by either the pressure of the containment or by a pump to the die. Other techniques for forming a melt containing the matrix and encapsulant can also be used. Essentially any of the techniques described in the prior art for forming a mixture of matrix and encapsulant can be used. On a continuous basis, the use of extrusion is preferred. When simple sugars are used as the matrix, the heat necessary to form the melt can be provided by the mechanical working of the screw alone or in cooperation with external sources of heat. Heated extruders for use in the food industry are well known and can be used for this purpose so that heat from both the external sources, such as the steam jacket around the extruder, as well as from the mechanical working of the extruder can be used.

When it is necessary to use a separate solvent to plasticize the matrix prior to introducing the encapsulant, the plasticizer/matrix melt may have its pressure reduced so as to vaporize a portion of the plasticizer. This reducing of pressure

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or venting to vaporize a portion of plasticizer may occur either before or after the encapsulate is introduced into the matrix into the melt when the encapsulate is of low volatility. If it is a highly volatile encapsulate then, the venting should occur  
5 prior to introduction of the high volatile component. After the highly volatile component is added, the melt is then extruded through a die and pressure cooled. Venting is particularly advantageous for use with encapsulates which are dissolved in a solvent which also function as plasticizers for the melts. Where  
10 both plasticizer and encapsulate are used and the matrix is soluble in both, the resulting solid product may have undesirable properties, such as tackiness, softness at low temperatures and a tendency to agglomerate. One technique for avoiding these problems is to simply use a total quantity of plasticizer and  
15 encapsulate which results in the desired properties. This procedure would restrict the loading of encapsulate which can be used. By venting the plasticizer, it is possible to incorporate higher quantities of encapsulate into the matrix without adversely affecting the properties of the final product.

20 When venting is used, it is necessary to repressurize the melt after the venting so as to eliminate from the melt any bubbles which might have been caused by venting of the solvent. In an extruder, this is easily accomplished using appropriate screw configurations. In other techniques, introduction of the

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melt into a melt pump after venting can accomplish the same purpose. The degree of repressurization depends upon the degree of pressure necessary to remove the voids which were formed in the matrix by the venting and be sufficient to allow extrusion through the die into the pressurized zone where cooling or solidification of the melt occurs.

While the foregoing discussion has presupposed that it is necessary to utilize a plasticizer and/or encapsulant to form the matrix melt, some matrices can be melted directly without the use of plasticizer and the encapsulate directly introduced into this melt. With such matrices, venting is not necessary. Further, when one is encapsulating an immiscible encapsulate, venting does not increase the total amount of encapsulate which can be incorporated into the matrix since the immiscible encapsulates have only a small effect upon the physical properties of the final product. In such instances, the removal of plasticizer is used primarily to control the properties of the final product. The use of large quantities of plasticizer tends to produce a softer and tackier product than reduced quantities of plasticizer in general. When the finished product is tacky, it may be overcoated with a material to reduce tackiness. Furthermore, in the case of a soft product, there is more of a tendency for the encapsulate to migrate to the surface and possibly to evaporate from the product. In such instances, it is possible to overcoat

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the product with a hard coating which prevents or reduces such migration and evaporation.

Figure 1 illustrates one method by which the process can be accomplished. In Figure 1, the matrix material is introduced  
5 into a continuous melter where it is melted. If necessary, the solvents described above will also be used to assist in the melting process. In the mixing zone of the melter 03, the injected encapsulate is mixed into the matrix. The matrix is then extruded and cooled to form the encapsulated product. The  
10 extrusion may be directly from the melting equipment under pressure or, as shown in Figure 1, a melt pump 06 may be employed to feed the extrusion die. In Figure 1, alternative methods are illustrated for cooling the encapsulated material. Discharge of the molten matrix/encapsulate mixture to atmospheric pressure  
15 illustrates the state of the art technique. For the embodiment of the current invention, the mixture of matrix and encapsulate is introduced into a pressure vessel, 08, where it is formed through a nozzle 09 into a continuous/batch pressure confinement. In this particular embodiment the pressure is provided by any  
20 gas, if necessary, food grade and/or inert, such as nitrogen, helium, or the like in pressure holding vessel 13. Pressure cooling is utilized wherein either the encapsulate contains a substantial quantity of volatile components, that is, components

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having boiling points substantially below the temperature of the melt.

After cooling under pressure, the product generally needs size reduction by grinding or the like to provide a free flowing material which is readily mixed with other components. If  
5 extruded, the nozzle utilized to extrude can be any type of nozzle and the size of the strands to be extruded is not critical. Typically, a "spaghetti" type nozzle will be employed so as to minimize the amount of particle size reduction which  
10 must be accomplished mechanically.

Numerous techniques exist in the plastics industry to chop or otherwise reduce in size long plastic strands for subsequent sale and use. Similar types of size reduction apparatus can be utilized in the present process. Some extruders have been sold  
15 where the face of the die is wiped continuously by knives to immediately reduce the exiting material to the desired size while plastic, and the thus divided material quenched in a suitable coolant. Such techniques can be applied in the present process as well.

20 An alternative method of recovering the product is to extrude the material into a pressurized mold and then allowing the material to solidify into a dense, nonporous mass. The mold can be cooled to assist in this process. In this particular embodiment, it would be preferable to employ injection molding

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type apparatus such as is well known in the plastics forming industry. In an injection molding apparatus, the molds are normally closed and the material injected under pressure and cooled before the mold is opened.

5       A further alternative is to introduce the melt under pressure into a body of liquid having a sufficient liquid head so as to establish a pressure at the point of melt introduction sufficient to preclude substantial volatilization of the volatile component. Essentially any liquid can be used for this purpose,  
10       however, food grade liquids are preferred. Alternatively, overriding gas pressure can be used over the body of liquid to assist in establishing the pressure at the point of melt introduction into the liquid body.

      In pressure cooling, the pressure is chosen to be  
15       sufficiently high so as to prevent foaming of the matrix if the matrix expands due to the vapor pressure of the plasticizer, solvent, or encapsulate. The amount of pressure necessary can be readily determined by simple experimentation. In the case of  
      volatile components, the pressure should be greater than the  
20       vapor pressure exerted by the volatile components at the molten product exit temperature. Many materials, e.g., the essential oils like orange oil, lemon oil and the like do not necessarily require pressure cooling since they tend to contain only small quantities of highly volatile materials. However, when these



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materials are enhanced with low boiling point top notes such as acetaldehyde, pressure cooling may offer advantages in reducing the microporosity of the finished product. The use of pressure cooling or atmospheric cooling with these materials is a matter  
5 of choice.

In an alternative embodiment, illustrated in Figure 2, the encapsulate is not introduced into the melter directly but rather is introduced either immediately prior to or into a static mixer into which the melted matrix ingredients are also introduced.  
10 The static mixer is illustrated as item 07, Figure 2. The remainder of the system is similar to that illustrated in Figure 1. In this embodiment, it is anticipated that the encapsulate in vessel 12, will be fed to a pressurized container, 04, and then pumped to the static mixer. However, the use of a  
15 pressurized container is dependent on the volatility of the encapsulate. In this embodiment, as in the previous embodiment, the plasticizer solvent can be vented from the system before the matrix and flavor components are admixed. Further, the melt pump, 06, can be omitted if the molten matrix is introduced  
20 directly from the continuous processor into the static mixer. In this embodiment, the encapsulates which are employed are typically those which have high solubility in the molten matrix, or disperse easily at the desired concentration level. In addition, this system also finds particular use when highly

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volatile components are to be encapsulated. The use of pump 05 and melt pump 06 facilitate the injection of low boiling point components into the molten matrix. The remainder of the process after the static mixer is the same as for the previous  
5 embodiment. Examples of products which can be encapsulated by this technique include fragrances, colors, flavors, pharmaceuticals and the like.

Another embodiment of the invention illustrated in Figure 3 is involved when encapsulating materials that are diluted in  
10 large amounts of volatile solvents that plasticize the matrix. When this is the case, the process would consist of an initial melting zone, a flavor mixing zone, a venting zone from which the solvent(s) are allowed to escape, followed by a re-pressurization zone and subsequent forming and cooling. Cooling could take  
15 place at either ambient or pressurized conditions, depending on matrix composition, process parameters, and encapsulate.

The equipment which can be used for this process can be essentially the same as that described above. In general, the solvents in which the materials to be encapsulated are dissolved  
20 are also solvents for the matrix materials. Thus, the use of a separate solvent in the formation of the melt is optional. However, the use of a separate solvent may be useful to eliminate losses of the desired components during the phase in which the solid matrix is being converted into a melt. The melt may be

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formed either in a batch process using a tank or large vat as discussed previously or through the use of extruder technology also as discussed previously. The melt is then vented at atmospheric pressure or under vacuum depending on the desired level of solvent removal, vapor pressure of the solvent itself, vapor pressure of the encapsulate, and molten matrix characteristics. The temperature is determined primarily by the conditions under which the venting of the melt is to occur and by the inherent vapor pressure of the solvent or solvents to be removed. If venting is accomplished to atmospheric pressure, higher temperatures are required than if vacuum conditions are used to vent. Once the melt has been vented to remove the desired quantity of solvent thereby concentrating the encapsulate, the matrix is repressurized so as to remove any voids which are formed during the venting and then formed through a die. The amount of solvent to be removed differs depending upon the matrix, the final properties desired in the solidified product, and loading. For hard, dense products more solvent must be removed than if the final product is to be soft. The product at this point may be either cooled under ambient pressure or under elevated pressure as described previously. Furthermore, once the matrix has been repressurized after venting, additional encapsulates may be introduced if desired. If these additional encapsulates are volatile, then it is preferred that the melt be

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extruded into a pressurized zone having sufficient pressure so as to preclude vaporization of significant quantities of the volatile components during solidification.

5 This technique has the advantage of allowing one to effectively concentrate vanilla solutions which have generally been difficult to concentrate because of the sensitivity of vanilla to degradation. It is believed that the matrix serves to stabilize the vanilla during the process.

10 These process steps are illustrated in one embodiment in Figure 3. Matrix materials are fed continuously to Melter 1 where they are melted prior to flavor injection. The matrix/flavor mixture is discharged to the feed port of Melter 2. Volatile solvents are vented out of the feed port of Melter 2, while the flavor containing melt is conveyed forward and  
15 discharged. In this embodiment, the material is fed to a melt pump which conveys the matrix/flavor mixture to forming and cooling operations. Of course, the melt pump is optional. Not shown in this illustration is the linkage of this process with pressure cooling which would be desirable in some cases.  
20 Flavorants which can be encapsulated in this technique include:

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<u>Flavor</u>	<u>Volatile Solvents</u>	<u>Wt % orig. flavor weight matrix</u>
Natural extracts	Water, ethanol	10 - 50%
Meat hydrolysates	Water	10 - 50%
5 Aqueous reaction flavors	Water	10 - 50%
Compounded flavors containing solvents	water, ethanol	10 - 50%

10 Additionally, the invention provides for a further enhancement of the above technique by a secondary injection of volatile encapsulates after venting of the solvent from the primary encapsulate and re-pressurization. This, especially when combined with the previously described pressure cooling, allows the encapsulation of a massive variety of encapsulate compositions.

15 A further variation on the above processes just described involves venting the melting equipment to remove the solvent which has been added to serve as the plasticizer before injection of the flavor component. Thus, if the solvent utilized is water, in case of continuous melting equipment, it would be arranged to  
20 have a first mixing zone where the matrix and water are intimately mixed, a second where heat and/or pressure are applied by any means to cause the matrix materials to melt/fluidize and then a pressure reduction section from which the water is allowed to vaporize and thus be removed. Re-pressurization of the matrix

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would follow, with subsequent flavor injection, mixing, forming, and finally cooling.

Figure 4 represents a generalized flow sheet for the foregoing embodiments. In its broadest aspect, the process involves converting the matrix materials into a melt, and mixing in the encapsulate and then cooling to produce a dense, amorphous product. When the encapsulate is not soluble in the matrix or is only slightly soluble, the result is an encapsulated product while if the encapsulate is soluble in the matrix material there results essentially a solid solution. In the preferred embodiments, a plasticizer solvent is introduced with the matrix to assist in melting. This plasticizer solvent may be vented if desired or may be retained in the mixture. The mixing of the encapsulate and matrix can occur either in a continuous process such as in a tubular reactor containing a helix screw to provide positive movement of the matrix from one end to the other or in a separate static mixture which is in fluid communication with the continuous melter which converts the matrix into a melt.

The foregoing process has the advantages of the prior art in that it is not limited to the use of a specific material. Prior art attempts to use maltodextrins as matrix materials have required the use of mixtures of oligosaccharides plus other materials to achieve successful melting and extrusion.

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Many of the matrix ingredients which are contemplated for use in the present process, are excellent film forming materials, such as maltodextrins, which tend to foam if extruded. By applying sufficient pressure in the pressure confinement to preclude foaming, a dense glassy matrix is achieved. Even  
5 matrices which do not naturally foam, will foam if the encapsulate contains substantial quantities of low boiling components such as acetaldehyde.

The materials which can be encapsulated will depend upon the matrix material chosen. By selecting the appropriate matrix, it  
10 is possible to encapsulate virtually any material with this particular technique. This includes insoluble, and slightly soluble encapsulates and also encapsulates which are soluble when the encapsulate does not detrimentally affect the plasticity and  
15 melting point of the matrix. Many matrix materials can be used in this embodiment. Indeed, prior art matrix materials such as those described in the United States Patent 5,009,900 as well as those disclosed in United States Patents 5,124,162, 4,879,130,  
4,820,534, 4,738,724, 4,707,367, 4,690,825, 4,689,235, 4,659,390,  
20 4,610,890, 4,388,328, 4,230,687, 3,922,354, 4,547,377, 4,398,422,  
3,989,852, 3,970,766, 3,970,765, 3,857,964, 3,704,137, 3,625,709,  
3,532,515, 3,041,180, 2,919,989, 2,856,291, 2,809,985, 3,041,180.

The classes of matrix materials include not only those listed in the above citations, but also materials such as mono-

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and disaccharides, oligomeric carbohydrates such as dextrans, and polymeric carbohydrates such as starches; soluble proteins and especially partially hydrolyzed proteins such as gelatin; other biopolymers; for example, hydrocolloids, gums, natural and modified celluloses; lipids, derivatives and/or any suitable mixtures of the above.

The choice of matrix composition is dependent upon the specific application and physical properties of the amorphous matrix and encapsulant. Levine and Slade (Water Science Reviews,

10 Volume 3, Chapter 2, "Water as A Plasticizer: physico-chemical aspects of low-moisture polymeric systems", pp 79-185, F. Franks (ed.), Cambridge University Press, 1988) reviewed the interrelationship between polymer molecular weight, process, and the role of water as a plasticizer in various food matrices. The physical attributes of glass matrices are key attributes in flavor encapsulation applications. A key requirement in matrix formulation is to control the plasticizer component of the matrix. While water is the most efficient agent for melt processing, the resultant matrix must remain in the non-rubbery state after flavor agents are incorporated. Therefore, one skilled in the art can choose from the variety of components listed in Table 1 as well as other ingredients generally available to the food technologist.



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TABLE 1  
POTENTIAL MATRIX COMPONENTS

1. High Molecular Weight Polymers

	<u>Proteins</u>	<u>Hydrocolloids</u>
5	Gelatin	Locust bean gum
	Casein	Glucans
	Lactalbumins	Guar gum
	Glutein/glutenin	Pectins
	Soy protein	Tragacanth
10	Myosin	Gum Arabic
	Actinomyosin	Carageenans
	Other soluble or meltable proteins	Alginates
		Inulins
15		Modified starches
		Pre-gelled starch
		Xanthan
		Gellan
		<u>Modified Celluloses</u>
		Methyl cellulose
20		Hydroxypropyl cellulose
		Hydroxypropyl methyl cellulose
		Sodium carboxymethyl cellulose (CM)

2. Intermediate Molecular Weight Compounds

25	Dextrins
	Corn syrup solids
	Cellulans
	Maltose syrup solids
	High fructose corn syrup solids

3. Low Molecular Weight Compounds

	<u>Plasticizers</u>	<u>Surfactants and Lipids</u>
30	Water	Polyglycerol esters
	Alcohols	Distilled monoglycerides
	Glycerol	Medium chain triglycerides
	Hydrogenated sugars	Lecithin
35	Sugars	Low molecular weight lipids
	Organic acids	

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Although not illustrated in the drawings, the finished product can be coated with an anticaking agent should that be necessary. However, caking is generally not a problem when the matrix materials have a sufficiently high softening point, typically above about 40°C. When the encapsulate is not soluble in the matrix, any encapsulate which remains on the surface of the finished product can be removed by utilization of suitable solvent in which the encapsulate is soluble but the matrix is either insoluble or only slightly soluble. While essentially any solvent having such characteristics can be utilized, food grade solvents having those characteristics are preferred. When the encapsulate is a lipophilic flavorant such as lemon oil, orange oil and the like, isopropanol has proven a successful solvent. Such washing may not be necessary where cooling has been accomplished by quenching in a quench medium selected to both cool and remove any surface flavorant from the product.

The present process allows for the successful encapsulation not only of high boiling point materials but also those having boiling points below about 100°C and most beneficially below 40°C in molten amorphous matrices. In the prior molten matrix encapsulation art, materials having boiling points below these limits have not been successfully encapsulated in concentrated form but only when diluted with other flavorants. For example, acetaldehyde may be somewhat successfully encapsulated when it

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has been introduced as a component in oil-based flavorants like lemon oil and orange oil. However, the present process provides for encapsulating pure acetaldehyde at high loadings above about 1 gram of acetaldehyde per 100 grams of matrix. Similar  
5 concentrations are possible with other low boiling point materials. With the low boiling point materials, the use of pressure cooling allows for the formation of a dense amorphous matrix, which may be known in the art as a glass; this material being substantially free of porosity, both gross porosity and  
10 microporosity. This substantial freedom from porosity will extend the shelf life of the product by reducing the amount of surface area exposed to the atmosphere. Thus, with low boiling point materials, the present process offers the advantage of increased loadings of materials in the matrix and a longer shelf  
15 life. The absence of porosity also ensures a dense material that will penetrate through the surface tension of liquids, expediting dissolution, and reducing the opportunity for lumping.

Further, the present process allows for the successful dense matrix encapsulation of materials diluted in volatile solvents.  
20 In the prior art, encapsulates diluted in volatile solvent systems could not be successfully encapsulated at commercially significant loads due to the plasticizing effect of the solvent on the matrix. This is overcome by the removal of the solvent after encapsulate injection via atmospheric or vacuum venting.

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Since the solvent removal takes place from the molten process stream, the resulting product is dense, thus the porosity formation caused by other solvent removal techniques such as spray or freeze drying is avoided. Additionally, secondary encapsulates may be injected into the process stream after removal of the primary encapsulate solvent. This is especially applicable to highly volatile secondary encapsulates, particularly when combined with the pressure cooling embodiment of the present process. Thus, the present process can successfully encapsulate a much wider range of materials in dense, amorphous matrices than was previously possible.

The present process when compared with spray drying and other state of the art processes, offers greater efficiency in encapsulating materials containing volatile components or those diluted in volatile solvents, often at a processing cost advantage. Furthermore, because essentially any material can be encapsulated by proper selection of processing conditions and matrix materials, a wide variety of products can be produced all having essentially about the same density and flow characteristics, an advantage in blending. Furthermore, products which have been encapsulated or otherwise incorporated into matrix materials can be blended together to produce unique flavor combinations with reduced concern for settling or stratification upon standing since the relative densities and particle sizes of

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the materials can be chosen to be approximately the same. Thus the present process will offer a full range of encapsulants all having approximately the same density and flow characteristics making handling, metering, measuring and the like much easier for the processor.

In the present description, the term "encapsulated product" includes not only those products truly encapsulated, where the encapsulate is insoluble in the matrix but also those products wherein the encapsulate is soluble in the matrix.

As can be appreciated from the foregoing description, the encapsulates in the present process do not need to be subjected to elevated temperatures in the presence of oxygen. This is a significant improvement over spray drying where the use of antioxidants is essential to be able to encapsulate products sensitive to oxidation. Such materials include but are not limited to citrus oils, highly unsaturated lipids, oxidation sensitive colorants and the like. The present process allows the encapsulation of such products reducing the need for the use of antioxidants.

In a preferred embodiment, the present invention provides a method for preparing compositions which contain one or more volatile aroma compounds, such as a coffee aroma compound or a volatile gas emitted during the baking of a dough product or the roasting of a food like nuts, peanuts, or meats, and which retain

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these compounds for a prolonged period of time. Specifically, the present method involves the formation of a composition in which the volatile aroma compound is encapsulated in a meltable food matrix, especially a food polymer. This embodiment may be carried out as follows:

(a) forming a melt comprising a volatile aroma compound and a matrix material; and

(b) solidifying the melt under a pressure sufficient to prevent substantial volatilization to directly obtain a dense, glassy matrix in which the volatile aroma compound is encapsulated.

In a particularly preferred embodiment, the volatile aroma compound is a coffee aroma compound. In the context of the present invention, the term "coffee aroma compound" means any volatile compound which confers the aroma of freshly ground coffee. The coffee aroma compound may be a single compound or may be contained in a mixture such as synthetic or natural coffee grinder gas or an aroma obtained by collecting the vapors removed from coffee extract during drying.

Examples of compounds which confer the aroma of freshly ground coffee include:

(i) the reaction products of methyl mercaptan with a molar excess of a mixture of carbonyl compounds which includes acetone, acetaldehyde, butyraldehyde and a lower alkane  $\alpha$ -diketone, as

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described in U.S. Patent nos. 3,852,481, and 3,873,746 which are incorporated herein by reference; and

(ii) 2-nonenal, 2-nonenol, lower alkyl acetals of 2-nonenal, esters of organic acids and 2-nonenol and mixtures thereof, as described in U.S. Patent no. 3,655,397, which is  
5 incorporated herein by reference.

In addition, any component of natural coffee grinder gas which confers the aroma of freshly ground coffee may be used. Such compounds include: ethyl mercaptan, ethyl acetate, methyl  
10 ethyl ketone, thiophene, pyridene, octane, 1-hexanol, and furfural. As noted above, these compounds may be used singly or in combination.

It is preferred that the coffee aroma compound be natural grinder gas. Grinder gas may be obtained by enclosing or hooding  
15 coffee grinding equipment, such as commercial grinders. The gases liberated from the ground coffee may be removed by a pump or rotary blower; additionally, when desired, a stream of inert, preferably moisture free, gas may be used to sweep gas from the coffee and to have the grinding operation take place in a  
20 substantially inert atmosphere. Such a process is described in U.S. Pat. No. 2,156,212 which describes a method of collecting gases evolved during roasting, but which can be equally applied to the collection of gases evolved during the grinding or cellular disruption of whole freshly roasted coffee beans. If

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pumping is employed, it may be desirable to cool the gas ahead of the pump so that the heat added by pumping will not deteriorate the aromatics contained in the gas.

5       The chemical composition of the evolved gas is largely carbon dioxide together with water vapor and the characteristic aromatic constituents of roasted coffee. The amount of moisture in the gas may be lowered by the use of dry roasting conditions and low-moisture quenches or quenching mediums. The evolved gas is preferably passed through a first condenser where it is cooled  
10   to between 35° and 50°F and where substantial quantities of water are removed. The relatively low-moisture gas is then fed to a second condenser, such as a jacketed, vertically-mounted, scraped-wall heat exchanger, which is cooled by means of a liquid gas refrigerant.

15       Preferably the second condenser is cooled by means of liquid nitrogen and the gas flow into the exchanger is maintained within the range of about 1 to 5 cubic feet per minute per square foot of heat exchanger surface. The nitrogen gas that evolves from the cooling system is useful as an inert gas stream which might  
20   be used elsewhere in the soluble coffee process, such as sweeping grinder gas from the grinder or inert gas packaging of a soluble coffee product.

      The aroma bearing gas is condensed into the form of a frost as it comes into contact with the heat transfer wall of the



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condenser. Typical grinder gas frost is collected at a liquid nitrogen jacket temperature of  $-195^{\circ}\text{F}$  to  $-220^{\circ}\text{F}$ , contains approximately 87% carbon dioxide, approximately 10% water, and approximately 3% coffee aromas. The frost, as it is removed from the condenser wall and collected, is thus very dilute in the coffee aromas. The frost may be held for a short period at low, such as liquid nitrogen, temperatures without deteriorating; however, it is preferred to immediately utilize the frost in accordance with the present invention.

10        Although the coffee grinder gas frost may be used directly as the coffee aroma compound, it is preferred that the coffee grinder gas frost be further process to increase the concentration of the compounds which confer the aroma of freshly ground coffee. Thus, the coffee grinder gas frost may be  
15        equilibrated at a pressure in excess of 750 psig to form three phases (a water phase, a liquid carbon dioxide phase, and a gaseous carbon dioxide phase), and the water drained, after which the liquid carbon dioxide is introduced into a vessel at a temperature less than  $-80^{\circ}\text{F}$ , and this vessel is vented at  $0^{\circ}\text{F}$  and  
20        then warmed to a temperature between  $0^{\circ}\text{F}$  and  $30^{\circ}\text{F}$  to obtain a highly concentrated liquid coffee aroma. This method is disclosed in U.S. Patent no. 4,574,089, which is incorporated herein by reference. A similar method for preparing a

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concentrated liquid coffee aroma is described in U.S. Patent No. 4,551,345, which is also incorporated herein by reference.

U.S. Patent no. 4,008,340, which is also incorporated herein by reference, describes a method for concentrating and  
5 stabilizing coffee grinder gas aroma, by combining coffee grinder gas frost with ascorbic acid or a salt thereof, warming to obtain a fluid, contacting the fluid with a liquid fluorinated hydrocarbon, separating the fluid condensate from the liquid fluorinated hydrocarbon, contacting the fluid condensate with a  
10 mixed solvent, and separating the condensed residue as an aqueous phase.

The matrix material can be any of those described above. Preferably, the matrix material is mannan oligomer obtained from spent coffee grounds. A suitable method for producing such  
15 mannan oligomers is described in U.S. Patent no. 4,508,745, which is incorporated herein by reference. Other preferred matrix materials are galacto-mannans, such as those obtained by multi-step coffee extraction by the application of heat/steam etc.

Depending on the exact identity of the coffee aroma compound  
20 being encapsulated and the identity of the matrix material, it may or may not be necessary to add a plasticizer to the matrix to form the melt. For example, when coffee grinder gas frost is used as the coffee aroma compound, the water contained in the frost may serve the role as plasticizer. Similarly, in synthetic

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grinder gases which contain liquids, the liquid may also serve to plasticize the matrix. When additional plasticizer is desired, a plasticizer such as water, glycerol, etc. may be added.

The need for and amount of additional plasticizer are easily  
5 determined by the skilled artisan by simple experimentation, keeping in mind that (i) it is desirable to form the melt at temperatures less than or equal to 275°F, preferably less than or equal to 225°F to avoid decomposition of the coffee aroma compound and/or the matrix material; and (ii) it is desirable  
10 that the final glassy matrix have a glass transition temperature,  $T_g$ , of at least 35°C, preferably at least 40°C.

The pressure under which the melt of matrix material and coffee aroma compound need be cooled will depend on the volatility of the coffee aroma compound but in general will be at  
15 least 500 psig, preferably at least 750 psig. The pressurized cooling of the melt is conveniently carried out in a closed chamber or vessel. Thus, the process may be carried out by extruding the melt directly into a closed chamber or vessel. The pressure in the cooling chamber or vessel may be created by an  
20 overriding pressure of an inert gas, such as nitrogen, air, etc. In a preferred embodiment, the cooling chamber is pressurized with a gaseous coffee aroma compound, especially coffee grinder gas.

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A key feature of the present method is that it affords dense, glassy matrices directly, without the need for an additional drying step, and the coffee aroma compound is stably encapsulated in the glassy matrix. That is, the present  
5 compositions will retain the coffee aroma compound for prolonged periods of time. Typically, the present compositions will contain the coffee aroma compound in an amount of from 0.01 to 10 wt.%, preferably 0.1 to 5 wt.%, based on the total weight of the glassy matrix.

10 Although it is preferred to encapsulate natural coffee grinder gas which has been treated to concentrate the aroma, it is also possible to formulate compositions which are a blend of two different glassy matrices, which each may contain a different coffee aroma compound and/or matrix material.

15 The present compositions which contain a coffee aroma compound may be added to any composition for which it is desired to enhance or create a coffee aroma. Thus, the present compositions may be added to any conventional instant coffee powder or ground roast coffee. The term "instant coffee" is  
20 meant to include not only those materials consisting of 100% coffee but also to substitute or extend coffees which may contain roasted grain, chicory, etc. The present compositions may also be added to instant cake mixes, instant pudding mixes, candies,

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or any other coffee flavored foodstuff which is stored in a dry state and is rehydrated when either prepared or eaten.

In another particularly preferred embodiment, the volatile aroma compound is a gas emitted during the baking of a dough product, like bread, or the roasting of a food, like nuts, 5 peanuts, or meats. The gas emitted during the baking of dough products may be collected in a similar manner as described for collecting grinder gas. Specifically, air or an inert gas such as nitrogen may be blown through the oven and then passed through 10 a series of cold traps to collect a bread baking gas frost, which can optionally be dewatered before incorporation in the matrix melt. Similarly, air or an inert gas such as nitrogen may be passed through the oven in which peanuts (either shelled or in-the-shell) are being roasted, and a peanut roasting gas frost 15 collected. In addition, the aroma released during the grinding of roasted peanuts may also be collected and used as the encapsulant. The aroma released when roasting true nuts, such as almonds and cashews, may be collected and used in a similar fashion. Other types of aromas such as the volatile gas emitted 20 during the roasting of meats such as poultry, beef, pork, lamb, etc. may also be collected and used as the volatile aroma compound.

The foregoing process and its variations are illustrated in the examples which follow. These examples are for illustration

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only and are not intended to limit the scope or application of the present process.

### EXAMPLES

#### Example 1

5 A carbohydrate based matrix composed of:

56% Amerfond (Domino Sugar, 95% Sucrose, 5% Invert sugar)

42% Lodex-10 Maltodextrin (American Maize, 10 DE)

2% Distilled monoglyceride (Kodak, Myverol 18-07)

10 was fed at a rate of approximately 114 grams/minute into the continuous processor (Figure 2) with water at 2 grams/minute. The mixture was melted in the processor. The processor was maintained at 121°C. The processor screws were operating at 120 RPM. The molten mixture was discharged directly to the melt pump. Acetaldehyde was injected into the molten matrix on the  
15 discharge side of the melt pump using a piston metering pump. A static mixer was used to blend the matrix and flavor together. Immediately prior to flavor injection the temperature of the molten matrix was approximately 138°C. The matrix and  
20 acetaldehyde mixture was then delivered under pressure to one of the nozzle discharges for forming and subsequent collection. The flow system was arranged so that forming and solidification could take place under either atmospheric or pressurized conditions. Four samples were taken:

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Sample 1: Ambient air cooled on trays.

Sample 2: Atmospheric pressure cylindrical collection vessel  
in ice bath.

5 Sample 3: Cooled in cold 99% isopropanol (initial  
temperature -18°C) at atmospheric pressure,  
approximately 130 g sample collected in 2000 g IP.

Sample 4: Pressure cooled; approximately 20 minutes under  
3275 kPa in a cylindrical collection vessel in an  
ice bath.

10 Visually, samples 1-3 were white and puffed with a porous  
internal structure. Sample 4 appeared dense, hard and relatively  
clear.

#### Analytical Results

	<u>Sample</u>	<u>% Acetaldehyde</u>	<u>Particle Density (g/cm<sup>3</sup>)</u>
15	1	.84	1.26
	2	.87	----
	3	.66	1.35
	4	1.67	1.63

#### Example 2

20 A carbohydrate based matrix composed of:

56% Sucrose Confectioner's sugar 6X (Domino Sugar)

42% Lodex Maltodextrin (American Maize, 10 DE)

2% Distilled monoglyceride (Kodak, Myverol 18-07)

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was fed at a rate of approximately 114 grams/minute into the continuous processor (Figure 1) with water at 2 grams/minute. The mixture was melted in the processor. The processor was maintained at 132°C. The processor screws were operating at 70 RPM. Diacetyl was injected into the molten mixture through a port in the continuous processor using a piston metering pump at a rate of approximately 10 grams /minute. After mixing the mixture was discharged directly into the Zenith melt pump. The matrix and diacetyl mixture was then delivered under pressure to one of the nozzle discharges for forming and subsequent collection. The flow system was arranged so that forming and solidification could take place under either atmospheric or pressurized conditions. Upon discharge from the melt pump, the product temperature was approximately 132°C. Four samples were taken.

Sample 1: Ambient air cooled on trays

Sample 2: Atmospheric pressure cylindrical collection vessel  
in ice bath

Sample 3: Cooled in cold 99% isopropanol (initial temperature -18°C) at atmospheric pressure, approximately 125 g sample collected in 2000 g IP (final IP temperature was -8°C).



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Sample 4: Pressure cooled; approximately 20 minutes under 2068 kPa in a cylindrical collection vessel in an ice bath.

Visually, samples 1-3 were pale yellow, relative opaque, and puffed with a porous internal structure. Sample 4 appeared dark yellow, dense, hard and relatively translucent.

Analytical results:

	<u>Sample</u>	<u>% Diacetyl</u>	<u>Particle Density (g/cm<sup>3</sup>)</u>
	1	2.40	1.33
10	2	2.26	----
	3	2.21	1.33
	4	3.97	1.49

Example 3

A carbohydrate based matrix composed of:

- 15        56% Amerfond (Domino Sugar, 95% Sucrose, 5% Invert sugar)  
         42% Lodex Maltodextrin (American Maize, 10 DE)  
         2% Distilled monoglyceride (Kodak, Myverol 18-07)

Flavor:

20        Vanilla extract (3 1/3 fold, 11.9% solids, 39.8% alcohol)  
was fed at a rate of approximately 114 grams/minute into continuous processor 1 (Figure 3). The mixture was melted in processor 1. Processor 1 was maintained at 143°C. Processor 1 screws were operating at 70 RPM. The vanilla extract was

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injected into processor 1 through a port at a flow rate of approximately 22 grams/minute. The molten mixture was discharged directly into processor 2 (143°C jacket temperature, 120 RPM).

Water and ethanol vapor were allowed to escape from the open

5 feedport of processor 2. The molten mixture was discharged into the melt pump which discharged through the nozzle onto trays for cooling and solidification. The product temperature exiting processor 1 was 102°C. The product temperature at the discharge of the melt pump prior to nozzle forming was approximately 115°C.

10 After cooling, the product was hard and dense, having the flavor characteristics of vanilla extract.

Analytical Results:

	<u>% Water</u>	<u>% Ethanol</u>
Initial composition		
15 (by mass balance)	10.3	6.4
Actual product composition	<u>6.4</u>	<u>&lt;.1</u>
Volatile solvent losses	3.9	6.4

Example 4

Conditions were as described in Example 3 except the feed rate  
20 for the vanilla was 30 grams/minute and no melt pump was used. The temperature out of processor 1 was 98°C and the product temperature out of processor 2 was 127°C.

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After cooling, the product was hard and dense, having the flavor characteristics of vanilla extract.

Analytical Results:

	<u>% Water</u>	<u>%Ethanol</u>
5 Initial composition		
(by mass balance)	12.4	8.3
Actual product composition	<u>7.3</u>	<u>&lt;.1</u>
Volatile solvent losses	5.1	8.2

Example 5

- 10 A carbohydrate based matrix composed of:

56% Amerfond (Domino Sugar, 95% Sucrose, 5% Invert sugar)  
42% Lodex Maltodextrin (American Maize, 10 DE)  
2% Distilled monoglyceride (Kodak, Myverol 18-07)

Flavor:

- 15 Natural beef flavor #12001 (Flavor and Food Ingredients, Inc., Middlesex, NJ) having 37.2% total solids and 14.6% salt.

Conditions were as described in Example 3 except the feed rate for the beef flavor was 29 grams/minute and no melt pump was used. The temperature out of processor 1 was 112°C and the

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product temperature out of processor 2 was 129°C. The jacket temperature was maintained at 160°C.

After cooling, the product was hard and dense, having the flavor characteristics of the original flavor.

5

Analytical Results:% Water

Initial composition

(by mass balance) 15.1

Actual product composition 7.0

10

Volatile solvent losses 8.1

Obviously, numerous modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described herein.

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WHAT IS CLAIMED AS NEW AND DESIRED TO BE SECURED BY LETTERS  
PATENT OF THE UNITED STATES IS:

1. A process for incorporating a volatile aroma compound into a matrix, said process comprising:

5 (a) forming a melt comprising said volatile aroma compound and a matrix material;

(b) solidifying said melt under a pressure sufficient to prevent substantial volatilization of said volatile aroma compound, to directly obtain a dense, glassy matrix having a T<sub>g</sub> of at least 35°C.

10

2. The process of Claim 1, wherein said melt is formed by:

(a) admixing a solid matrix material with a plasticizer and heating to form a melt;

(b) reducing the pressure on said melt so as to remove any gases present and to volatilize at least a portion of said plasticizer and removing said portion from said melt;

15

(c) admixing with the resulting melt with volatile aroma compound under pressure and temperature conditions sufficient to prevent substantial volatilization of said volatile aroma compound.

20

3. The process of Claim 1, wherein said volatile aroma compound is a coffee aroma compound, a volatile gas emitted when baking dough, or a volatile gas emitted when roasting a food.

4. The process of Claim 3, wherein said volatile aroma compound is a coffee aroma compound.

25

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5. The process of Claim 4, wherein said coffee aroma compound is immiscible in said matrix.

6. The process of Claim 4, wherein said coffee aroma compound is soluble in the matrix.

5 7. The process of Claim 4, wherein said coffee aroma compound is natural coffee grinder gas.

8. The process of Claim 4, wherein said coffee aroma compound is synthetic coffee grinder gas.

10 9. The process of Claim 4, wherein said coffee aroma compound is obtained by collecting aroma vapors from coffee extract during drying.

10. The process of Claim 4, wherein said matrix material is selected from the group consisting of maltodextrins, corn syrup solids, maltose syrup solids, high fructose corn syrup solids, 15 starches, hydrocolloids, gums, proteins, partially hydrolyzed proteins, modified proteins, modified hydrocolloids and modified celluloses.

11. The process of Claim 4, wherein said matrix material comprises a mannan oligomer.

20 12. The process of Claim 11, wherein said mannan oligomer is obtained by hydrolyzing spent coffee grounds.

13. The process of Claim 4, wherein said matrix material comprises a galacto-mannan.

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14. The process of Claim 4, wherein said solidifying is carried out in a closed chamber under a pressure of 500 to 750 psig.

5 15. The process of Claim 14, wherein said chamber contains coffee grinder gas.

16. The process of Claim 3, wherein said food is a nut, peanut, or meat.

17. A process for incorporating a volatile aroma compound into a matrix, said process comprising:

10 (a) admixing a solid matrix material with a plasticizer which comprises a flavor compound dissolved in a volatile solvent and heating to form a melt;

(b) venting said melt at either atmospheric or reduced pressure so as to remove any gases present and to volatilize at  
15 least a portion of said volatile solvent and removing said portion from said melt;

(c) admixing with the resulting melt a volatile aroma compound under pressure and temperature conditions sufficient to prevent substantial volatilization of said volatile aroma  
20 compound, to obtain a volatile aroma compound-containing melt; and

(d) solidifying said volatile aroma compound-containing melt under a pressure sufficient to prevent substantial volatilization of said volatile aroma compound, to directly obtain a dense,  
25 glassy matrix having a Tg of at least 35°C.

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18. A dense, amorphous solid comprising a matrix and a volatile aroma compound encapsulated in said matrix.

19. The dense, amorphous solid of Claim 18, wherein said volatile aroma compound is a coffee aroma compound, a volatile gas emitted when baking dough, or a volatile gas emitted when  
5 roasting a food.

20. The dense, amorphous solid of Claim 19, wherein said volatile aroma compound is a coffee aroma compound.

21. The dense, amorphous solid of Claim 20, wherein said  
10 coffee aroma compound is coffee grinder gas.

22. The dense, amorphous solid of Claim 20, wherein said coffee aroma compound is obtained by collecting aroma vapors from coffee extract during drying.

23. The dense, amorphous solid of Claim 20, wherein said  
15 matrix comprises a mannan oligomer.

24. The dense, amorphous solid of Claim 23, wherein said mannan oligomer is obtained by hydrolyzing spent coffee grounds.

25. The dense, amorphous solid of Claim 20, wherein said matrix comprises a galacto-mannan.

20 26. The dense, amorphous solid of Claim 25, wherein said galacto-mannan is extracted from coffee.

27. The dense, amorphous solid of Claim 18, wherein said food is a nut, a peanut, or a meat.





FIG. 2

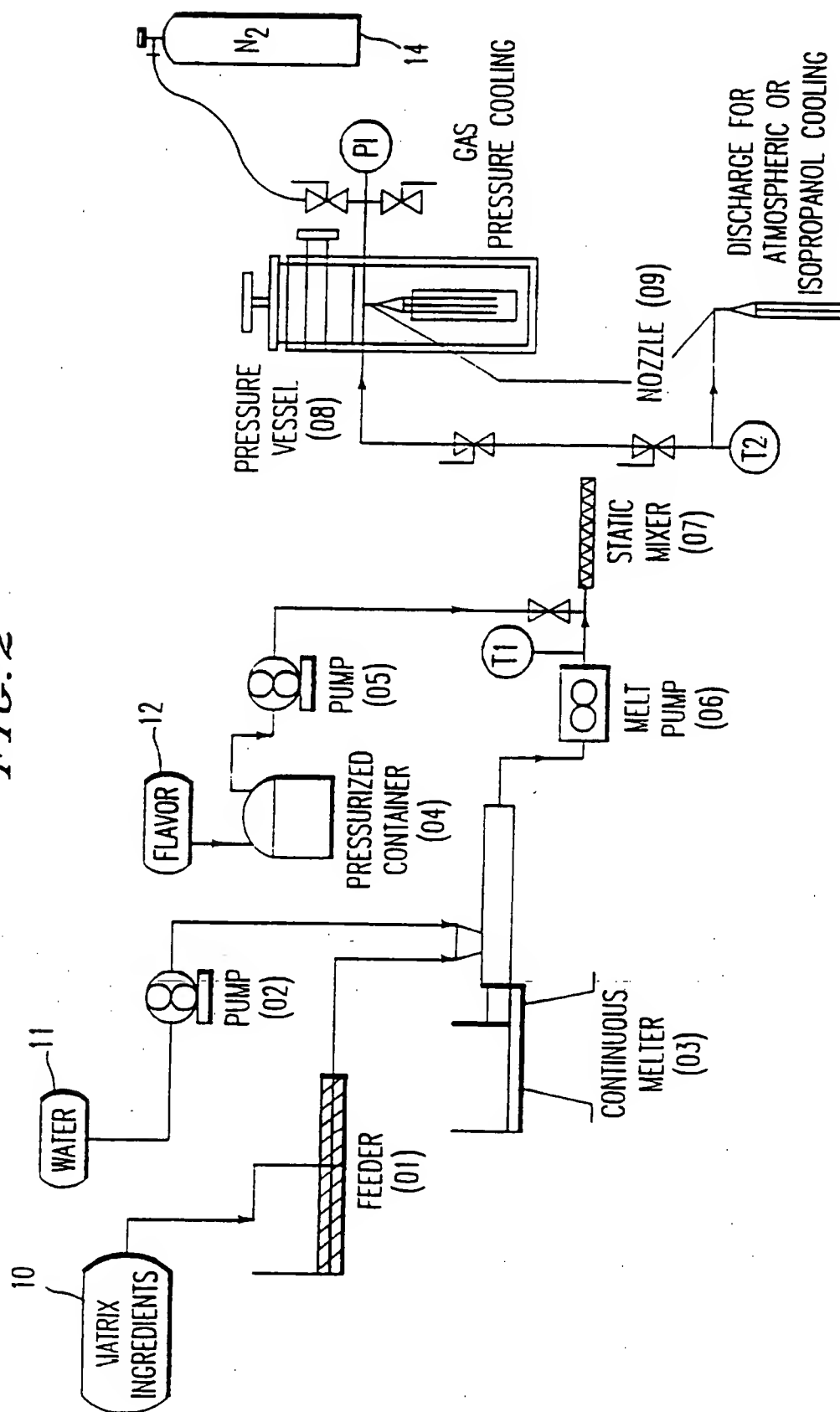


FIG. 3

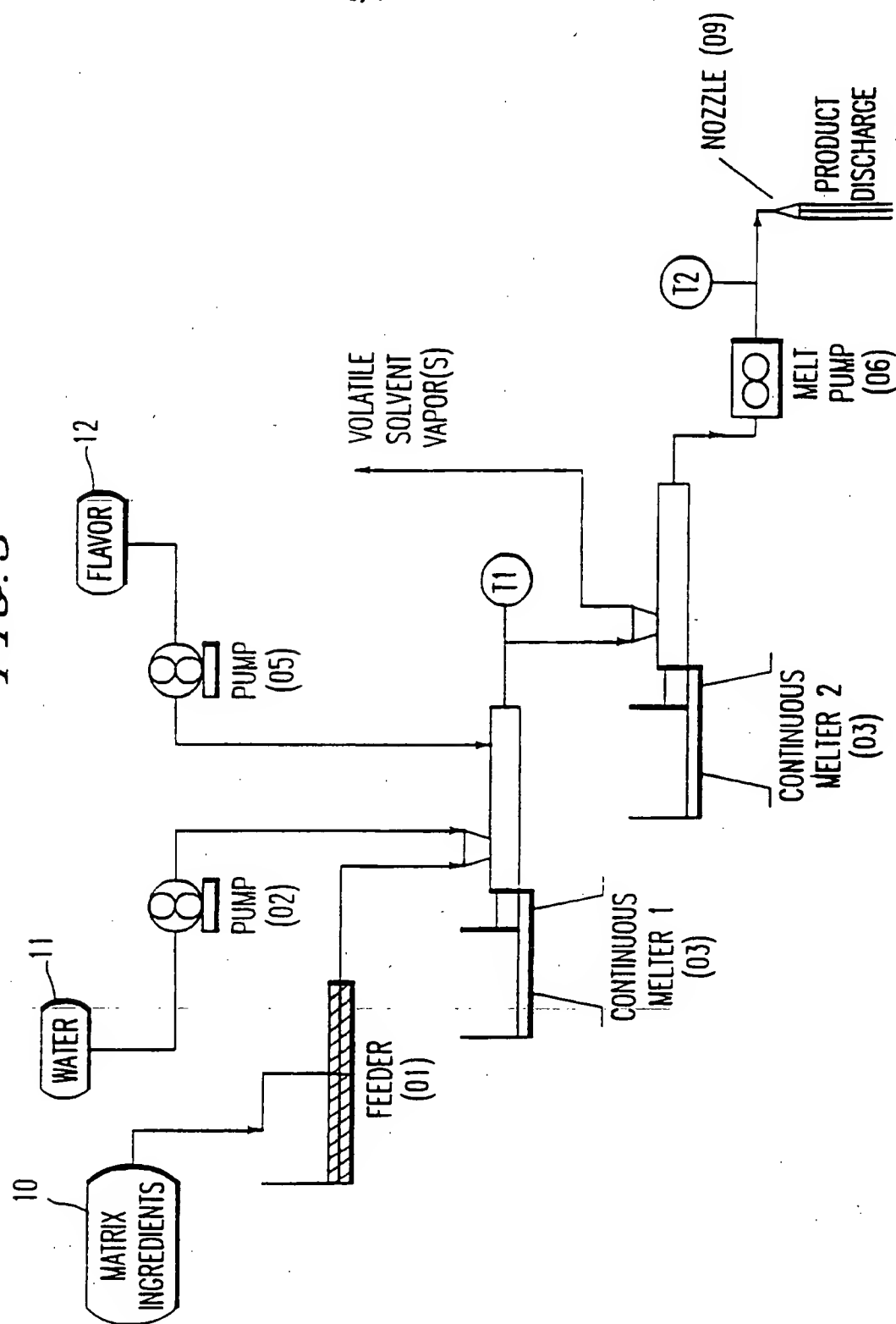


FIG. 4

